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## **Composite Propellants with Bi-Plateau Burning Behaviour**

Kym M. Ide

DSTO-GD-0344

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# Composite Propellants with Bi-Plateau Burning Behaviour

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DSTO-GD-0344

## ABSTRACT

The literature describing the mechanism of combustion in composite rocket propellants exhibiting bi-plateau burning has been reviewed. A wide distribution of AP particle size in a definite coarse/fine ratio is required to ensure adequate interstitial spacing resulting in abnormally depressed burning due to interference of the fine AP fraction combustion by the binder melt layer. Titanium dioxide added to the formulation acts to increase the binder melt layer viscosity restoring normal burning at low and high pressures, leaving abnormal burning and a plateau or mesa effect at intermediate pressures. The use of dimeryldiisocyanate curative produces a unique plateau at lower pressures due to increased binder liquefaction. Formulation variables can be adjusted to tailor the pressure levels and burn rates of the plateaus formed.

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# Composite Propellants with Bi-Plateau Burning Behaviour

## Executive Summary

For a typical composite propellant the burning rate is proportional to the pressure raised to an exponent  $n$ ; an exponent in the range 0.25 – 0.6 is usual. An increase in the burning surface produces an increase in operating pressure. Hence, the burning rate and pressure exponent have a significant effect on the design of a rocket motor. A propellant possessing a zero ("plateau burning") or negative pressure exponent would be considered advantageous in certain rocket motor systems where insensitivity of burn rate to pressure can have benefits for motor case design.

In a "boost-sustain" motor where a high level of thrust is required during a launch phase and lower but extended thrust is required during the "sustain" operation, control of thrust output is highly desirable. Some applications may call for more than one boost phase and more than one sustain phase. For example, it may be desirable to boost the rocket motor to a faster speed or higher altitude after the sustain phase. In a solid propellant rocket motor such multiphase operation may be achieved by the use of bi-plateau propellant.

In addition, bi-plateau propellants are also capable of providing higher ratios of boost to sustain operating pressures (turndown ratio) than systems employing multiple propellants, which may be beneficial in some applications requiring boost-sustain operation. A single propellant capable of producing stable output at multiple operating pressures would be simpler and more reliable than rocket motors containing propellant grains with multiple propellant formulations or complex mechanical assemblies.

An understanding of the combustion processes that cause plateau and/or bi-plateau burning would provide the means to tailor the burn-rates by varying the propellant formulation. The burn rates and the pressures at which they operate could be selected to achieve a desired performance goal for the specific motor during its operation.

This paper reviews the literature available to the author up to August 2001 describing the mechanism of plateau burning in composite rocket propellants.

The existence of binder melt layer interference with combustion of the fine AP fraction has been established as the mechanism leading to plateau burning. Titanium dioxide is included to increase the binder melt viscosity and restore normal burning at low and high pressures leaving abnormal burning and a plateau effect at intermediate pressures. Use of a specific curative leads to an additional plateau at low pressure and bi-plateau burning behaviour.

Establishing a knowledge base for composite rocket propellants capable of providing multiphase operation with greater thrust and manoeuvrability during the end game will allow the provision of better advice to the ADF on the propellant aspects of these types of systems.

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# 1. Introduction

The relationship between operating pressure and burning rate in a solid propellant can be expressed mathematically as:

$$r_b = ap^n$$

where  $r_b$  is the burning rate,  $p$  the absolute pressure,  $a$  an empirical constant and  $n$  the pressure exponent[1]. For a typical composite propellant the pressure exponent is in the range 0.25 – 0.6, an increase in the burning surface produces an increase in operating pressure[2]. Hence, the burning rate and pressure exponent have a significant effect on the design of a rocket motor. A propellant possessing a zero ("plateau burning") or negative pressure exponent would be considered advantageous in certain rocket motor systems where insensitivity of burn rate to pressure can have benefits for motor case design[3].

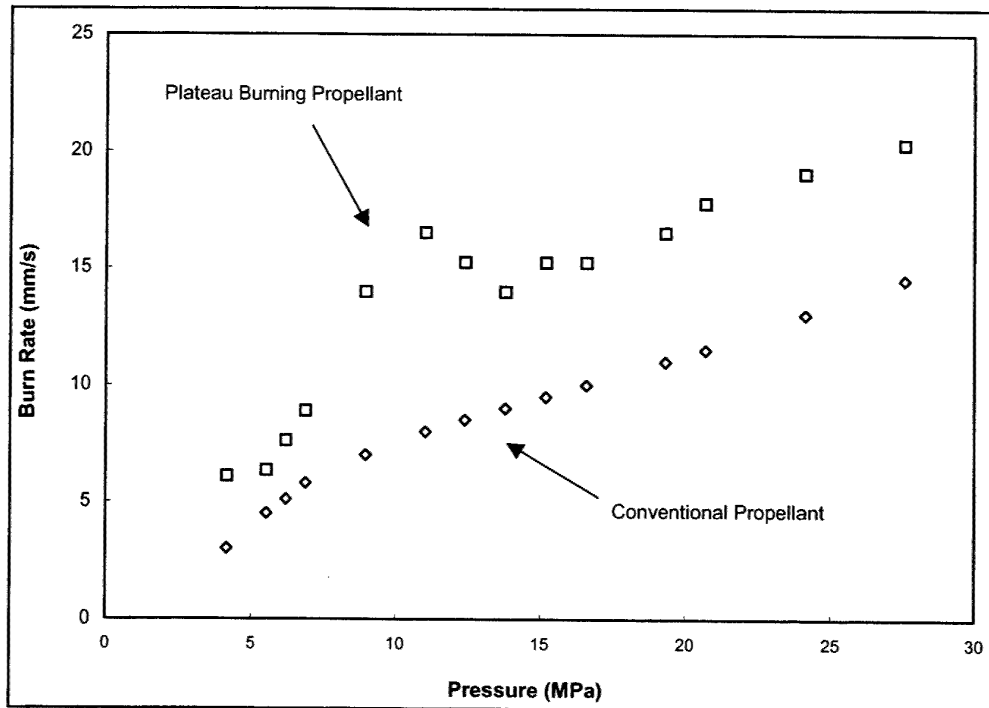


Figure 1 Chart of typical burn rate data for plateau burning and conventional propellants.

Tailoring of the propellant formulation can provide plateau burning over a wide pressure range anywhere between 0.7 and 48 MPa with burn rates from 5 to 50 mm.s<sup>-1</sup> which offers benefits in the approach to energy management [3, 4]. For the same maximum operating pressure, a propellant with reduced sensitivity of burn rate to operating pressure over a wide pressure range (plateau) allows a higher average chamber pressure to be maintained

over the burn time compared to a conventional propellant (see Figure 2), this leads to increased performance. Alternatively, for the same value of average chamber pressure for either a plateau burning or conventional burning propellant, a lower maximum operating pressure can be employed for the plateau burning propellant. Results of a design study which illustrate this can be seen in Table 1[5]. A motor with higher propellant loading and mass fraction can be achieved with a simple centre perforate (CP) grain design for boost-sustain[6]. Weight reductions arise from a thinner motor case design.

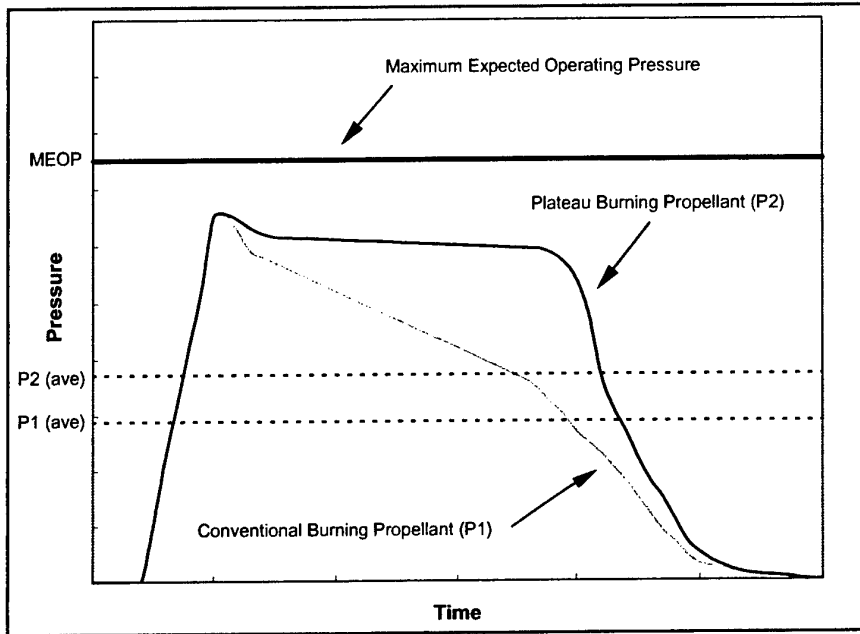


Figure 2 Chart showing higher average chamber pressure for plateau burning propellant



Table 1. Design Analysis for Single Operational Pressure Motor

Propellant	Conventional	Plateau	Plateau
Plateau Option	N/A	1	2
MEOP* (MPa)	17.2	17.2	11.5
$P_{\max}$ (MPa)	13.5	16.1	10.8
$P_{\text{avg}}$ (MPa)	9.7	14.5	9.7
$I_{\text{sp}}$ (s)	245.8	256.7	245.8
Case Weight (kg)	29.1	27.1	19.3
Mass Fraction	0.736	0.743	0.777

In a "boost-sustain" motor where a high level of thrust is required during a launch phase and lower but extended thrust during the "sustain" operation control of thrust output is highly desirable [6]. Some applications may call for more than one boost phase and more than one sustain phase. It may be desirable to boost the rocket motor to a faster speed or higher altitude after the sustain phase allowing greater manoeuvrability and speed during the "end-game". In a solid propellant rocket motor this may be achieved by the use of multi-phase or bi-plateau propellant and a means of controlling the rocket motor chamber pressure allowing switching between each operating pressure range.

In addition, bi-plateau propellants are also capable of providing higher ratios of boost to sustain operating pressures (turndown ratio can be improved from less than 2:1 to more than 5:1[5]) than systems employing multiple propellants, which may be beneficial in some applications requiring boost-sustain operation[6]. A single propellant capable of producing stable output at multiple operating pressures would be simpler and more reliable than rocket motors containing propellant grains with multiple propellant formulations or complex mechanical assemblies.

An understanding of the combustion processes that cause plateau and/or bi-plateau burning would provide the means to tailor the burn rates by varying the propellant formulation. The burn rates and the pressures at which they operate could be selected to achieve a desired performance goal for the specific motor during its operation.

The literature was reviewed to discover the current state of knowledge of the combustion processes leading to plateau and/or bi-plateau burning in composite propellants. The discussion will necessarily concentrate on propellant formulations using hydroxy-terminated polybutadiene (HTPB) binder and ammonium perchlorate (AP) oxidiser as

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\* Maximum Expected Operating Pressure

these widely used ingredients are included in most of the formulations of plateau burning propellants commonly reported.

## 2. Mechanism for Plateau Burning in Composite Propellants

### 2.1 Wide Bimodal Oxidiser Particle Size Distribution

Price et al.[7] summarise some early studies[8, 9] conducted in the 1960's of the burning behaviour of ammonium perchlorate-hydrocarbon (AP/HC) binder propellants with monomodal oxidiser size distribution where plateau burning was detected. In these studies the independence of burning rate on pressure in a mid-pressure (7-10 MPa) range was reported as "anomalous" burning. Some propellant formulations exhibited decreasing burn rate ("mesa") with increasing pressure to the extent that in some cases burning could not be sustained. It was observed that the anomalous behaviour was sensitive to the choice of AP particle size (generally fine particles range from 2 to 5  $\mu\text{m}$  and coarse 150 to 400  $\mu\text{m}$ ), binder type and oxidiser/binder ratio (varying from 65/35 to 90/10)[3]. The abnormal or "anomalous" burning was associated with localised intermittent burning at shifting sites on the burning surface, this phenomenon will be discussed in more detail below.

Another early study by Jacobs and Whitehead[10] recognised the failure of the granular diffusion flame (GDF) model[11]:

$$\frac{1}{r} = \frac{a}{P} + \frac{b}{P^{1/3}} \text{ where } r = \text{burn rate, } P = \text{pressure, } a = \text{constant, } b = \text{constant,}$$

to predict the independence of burn rate and pressure when conditions described as favouring plateau burning occur. The important factors included propellants with small AP particles, low AP content or fuels that readily melt.

Later investigations [12, 13] included propellants with increased specific impulse compared to the fuel rich propellants of the earlier studies. Higher solids loadings were achieved in formulations by using bimodal oxidiser particle size distribution. Propellants exhibited abnormal burning behaviour when specific combinations of coarse and fine oxidiser particle sizes were employed. The behaviour was present only for a limited range of AP coarse/fine ratios in which the two particle sizes were widely different and the size of the fine AP was small ( $\leq 20 \mu\text{m}$ ).

In propellants with high solids loading the presence of a wide distribution in oxidiser particle size and coarse and fine AP in specific proportions has been recognised as the precondition for production of a plateau burning propellant and subsequently bi-plateau burning propellant[12, 14, 15].

Price [7] proposed a simplified view of the combustion zone in a bimodal propellant. The burning surface consists of a mixture (or "matrix") of fine AP and binder which contains and surrounds an array of coarse AP particles. Propellant combustion depends on the interaction between the burning matrix and coarse AP. The matrix burns with a premixed flame, which interacts with the burning coarse AP near the surface of contact with the coarse particles, but may not be capable of sustaining combustion on its own. The burning of the coarse particles involves fuel from the matrix "vapours". The extent of the interaction was related to how well the matrix will burn on its own and how far apart the coarse particles were [14, 16, 17]. The major contribution or control of the propellant burning rate by the matrix formulations was regarded as responsible for the plateau burning of bimodal propellants. A number of variables were identified as having an important influence on burn rate; the coarse/fine ratio (which affects the coarse particle spacing), relative size of coarse and fine AP, oxidiser/fuel ratio in the matrix and binder type. It was noted that lateral heat transfer exists in the condensed phase and under some conditions flow of binder melt onto the coarse AP surfaces occurs.

## 2.2 Significance of the Binder Melt Layer

Studies describing the importance of the condensed phase reactions in the combustion of HTPB/AP propellants have been conducted by many workers [18, 19, 20, 21, 22]. The formation of a melt layer due to binder pyrolysis was linked to the magnitude of the burn rate. In the specific case of a propellant with wide oxidiser size distribution it has been reported that the presence of a binder melt layer is necessary for the development of abnormal burning [7, 10, 12, 14, 15, 23, 24, 25].

Abnormal burning of wide-distribution AP particle size propellants results from a binder melt layer interference mechanism [7, 12, 25]. When the ratio of coarse to fine AP is increased to a level where the space between coarse particles becomes sufficient, combustion of the suitably fine AP particles can be inhibited by the binder melt layer [25, 26]. This produces depressed burning, in extreme cases the binder/fine AP matrix may fail to support burning [10]. The depression in the burning rate becomes a maximum at intermediate pressures as a result of a balance between the thickness and viscosity of the melt layer.

Chakravarthy et al. [24] and Price et al. [7] studied the melting behaviour of hydrocarbon binders in a hot stage microscope. The binder systems (polybutadiene-acrylonitrile-acrylic acid terpolymer, PBAN, isophorone diisocyanate, IPDI-cured HTPB and dimeryl diisocyanate, DDI-cured HTPB) differed markedly in melt temperature and viscosity. PBAN binder melted at 480°C and vaporised almost simultaneously at 500°C (defined as a "low melt" binder). For HTPB binders the melt characteristics varied with curative used. DDI-cured HTPB melted at 260°C to a low viscosity fluid (defined as a "high melt" binder). As the temperature approached 500°C the melt started to bubble. IPDI-cured HTPB melted slowly over a range of temperatures from 330-370°C. Both decomposed over a 10°C temperature range around 500°C.

The DDI-cured HTPB binder melt is thicker and more viscous at lower pressures due to lower temperatures of combustion at the surface [24, 7]. Cohen [27] suggested that as pressure increases the combustion flame is forced closer to the surface causing greater heat feedback and increased surface temperature, the melt becomes less viscous and is able to flow (as discussed in [7]). Normal burning is restored at higher pressure as the melt becomes too thin to inhibit burning in the ultrafine AP/binder matrix [25]. At extremes of high and low pressure, the burn rates and the pressure exponents of the wide oxidiser size distribution propellants are equivalent to those of similar normal burning propellants [7].

Xu et al. [28, 29] describe a model for combustion which they state takes into account initial temperature and AP particle size to explain the plateau, mesa and normal burning behaviours. The description of the burning mechanism involves the division of the burning surface into two regions (see Figure 3). One region consists of AP covered by molten binder (region I) and the other, uncovered AP and surrounding binder (region II). The two regions are described as obeying completely different combustion rules, with the overall combustion depending on the interaction between region I and II.

In region I the combustion is controlled by the condensed-phase reactions and opposed gasification of AP. The gasified AP molecules cannot rapidly enter the flame zone and must pass through the molten binder layer even at low pressure. The thickness of the molten binder layer depends on its fluidity and the burning rate in region I. As the pressure increases the condensed-phase reactions in AP decrease [30] and the rate of gasified AP molecule collisions increase. The opposed gasification increases ensuring a negative pressure exponent when pressure exceeds a certain value. Combustion in region II is controlled by the AP decomposition and presents as normal burning with a positive pressure exponent.

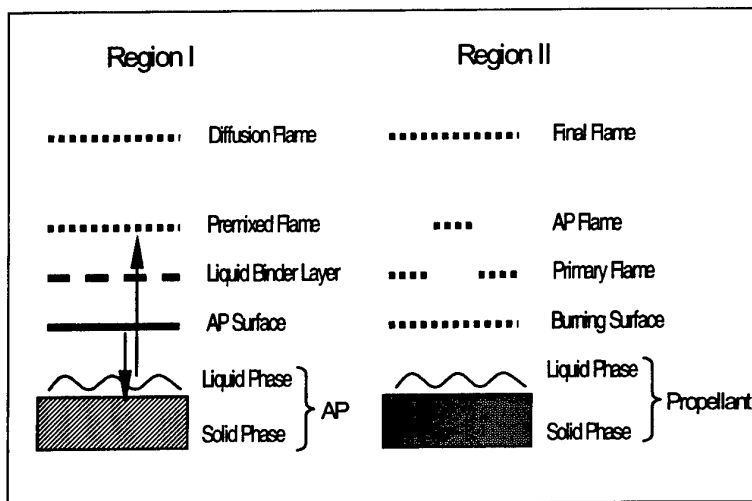


Figure 3 One Dimensional Model of Burning Mechanism Proposed by Xu[28]

The fraction of burning surface existing as region I depends on the fluidity of the molten binder layer, pressure, AP particle size, content of metal fuel and content and type of burning rate catalysts. Any factors acting to increase the fraction of the burning surface existing as region I favours a negative pressure exponent (mesa) for the propellant combustion.

The importance of melt layer viscosity in the production of abnormal burning has been reported [7, 12, 25]. The viscosity of the melt layer may be lowered by addition of a plasticiser which subsequently improves the plateau [3]. The higher volatility of the plasticiser may contribute to the restoration of normal burning at high pressure.

Jojic and Brewster [15] argued that although the role of fine AP in composite propellants with wide-distribution AP particle sizes was recognised as significant for the production of abnormal burning, the combustion mechanism in the matrix was not well defined. They studied the condensed phase reactions in mixtures of fine AP and HTPB at high heating rates by laser irradiation. An exothermic event was observed between 250 and 300°C where none existed in the decomposition of binder alone. It was suggested that the condensed-phase chemical interaction between fine AP and HTPB plays a significant role in the combustion.

Price [7] argued that "anomalous" burning described in an early study[8] had not received adequate consideration due to the growing favour of the melt flow interruption mechanism. It was suggested that AP particles gasify faster than binder in the mid-pressure regime, causing local areas of the burning surface to be depleted of AP. Until new particles were exposed burning would have to be sustained in adjacent areas. Uneven surface regression leading to localised intermittent burning would reduce the net heat flux to the surface and hence the average burning rate. Price concluded that it was clear that propellants with "high melt" binder (HTPB) were more prone to plateau burning. Instances of plateau burning reported in propellants with "low melt" binder (PBAN) may have been as a result of intermittent burning.

### 2.3 Burn Rate Modifier for Plateau Burning

The inclusion of a burn rate modifier or catalyst can further promote (and allow tailoring) or eliminate the production of plateau ballistics.

Various workers have observed the effect of binder melt flow on addition of a burn rate catalyst to AP/HTPB propellant. Handley and Strahle [31] found a significant physical or chemical effect involving removal or inhibition of the binder melt flow at low pressures. The "catalytic" mechanism could not be explained, however, on addition of ferrocene or iron blue the deflagration rate increased. Fong and Hamshire [32, 33] and Fong and Smith [12] investigated the effect of copper chromite on the plateau burning behaviour of composite propellants. The catalyst was found to enhance binder decomposition and plateau burning behaviour as a result of increased binder melt at low pressures. The binder melt layer would be thicker at low pressure and have longer residence times and

consequently catalysed condensed phase reactions would have a greater chance of occurring.

Yin et al. [34] reported the effect on the combustion mechanism of adding a burn rate catalyst to AP/polyurethane (PU) propellants with negative pressure exponents. Calcium carbonate ( $\text{CaCO}_3$ ) was added to one formulation and the combustion surface examined using high-speed cinemicrophotography and scanning electron microscopy (SEM). Molten binder was observed to cover a fraction of the burning surface in both mesa and non-mesa pressure regimes. A reaction scheme was proposed involving  $\text{CaCO}_3$  and AP which competes with the decomposition of AP. The effect increases at higher pressures further inhibiting the AP burning. The increased mobility of the  $\text{CaCO}_3$  in the binder melt and the depressed burning from melt layer interference causes enhancement of the mesa effect and the size of the plateau is increased.

Refractory oxides such as titanium dioxide, aluminium oxide, zirconium dioxide and silicon dioxide are also used as burn rate modifiers. Titanium dioxide is the preferred choice due to its superior ability to tailor the burn rate and operating pressure for plateau burning in a wide AP particle size distribution propellant formulation [3, 6, 35]. It is also abundant and therefore economical. Titanium dioxide promotes a plateau in the pressure range from 5 to 10 Mpa [26, 35, 36]. The plateau indicates the transition from normal burning to the depressed burning inherent in wide AP particle size distribution propellants to normal burning at the end of the plateau.

Price [37] described the effect of titanium dioxide on a HTPB-DDI binder system in a hot stage microscope. When heated gradually to 500°C it caused reduced fluidity and coagulation of binder melt into wet clumps with surrounding liquid. In propellants with a wide AP particle size distribution very fine particles of titanium dioxide act to increase the binder melt layer viscosity such that no interference with the fine AP particles occurs and normal burning is restored [26]. Titanium dioxide has been used to even out pressure exponents [38] and is reported to not act as a burn rate catalyst [39, 40, 41, 42].

Brill and Budenz [36] state that the mechanism causing plateau burning is not agreed upon and refer to thermogravimetric analysis (TGA) which suggests some additional chemical reactivity is introduced by titanium dioxide [26] (refer below). In flash pyrolysis experiments of AP-HTPB mixtures they report an accelerated rate of evolution of gaseous products when 5% titanium dioxide was added.

They concluded that titanium dioxide was a positive catalyst for decomposition but at experimental pressures well below those where plateau burning is observed. Therefore no relationship to the mechanism for plateau burning and this catalytic behaviour can be assigned. It was speculated that if the same observations are valid at higher pressures then titanium dioxide may dampen the amount of heat released and contribute to a reduction of the burning rate.

Freeman et al. [26] investigated the contribution of the monomodal AP-Hydrocarbon (HC) matrix to plateau burning of propellants with bimodal AP particle size distributions.

Combustion photography (video) revealed when the matrix would, and would not, sustain burning as AP particle size and pressure were varied (referred to as burn/no-burn domains). Burning was more difficult to sustain as the amount of melt layer present increased. HTPB-DDI cured matrices with the highest degree of melt had a no-burn region that extended over nearly the entire range of pressures used, HTPB-IPDI had a mid-pressure no-burn region and PBAN showed no significant no-burn region. These effects were more evident for finer AP particles. When ultrafine titanium dioxide ( $0.02\ \mu\text{m}$ ) was added the no-burn domains were eliminated and burning rates increased. When coarse titanium dioxide ( $0.5\ \mu\text{m}$ ) was added a no-burn domain in the mid-pressure region occurred for the PBAN matrix and a mesa was produced in the burn rate curve for the HTPB-IPDI matrix.

The authors question why a no-burn domain was present in the "low melt" PBAN matrix after the addition of titanium dioxide. Titanium dioxide supposedly acts to restore normal burning, only by suppressing the melt flow. In the case of coarse titanium dioxide the particle size would be of the same order as the ultrafine AP (UFAP), they questioned its capacity to prevent the flow of the melt layer over these particles. It was speculated that titanium dioxide has some effect in the gas phase reactions or a catalytic effect on decomposition at the surface. Evidence to support this came from TGA of propellants which showed an increase in the rate of mass loss in the presence of titanium dioxide and surface micrography of quenched samples where no evidence for inhibition by the melt layer was detected. It was argued that the accumulated debris observed supported the assumption of surface reactions aided by titanium dioxide. However, the surface micrography of quenched samples was only available at one combustion pressure (1000 psi) which did not appear to coincide with the plateau in the burn rate data. They concluded that no models have been developed to convincingly explain the mechanisms leading to plateau burning or the burning rate increase on addition of titanium dioxide.

Boxx et al.[25] compared features on the surface of extinguished plateau burning propellants. Electron dispersive spectroscopy (EDS) and SEM techniques were used to confirm that at low pressure, clusters of the burn rate modifier (not binder char) are present on the surface of the coarse AP particles. The fine AP/binder matrix and coarse AP burn with comparable rates. In the plateau region it was observed that fine AP was covered by a binder melt layer. The conclusion was that this caused suppression of the fine AP and accounted for the plateau.

The features of a propellant which did not contain burn rate modifier showed no material was present at the centres of the coarse AP, confirming that clusters of additive collect there in formulations containing it. The greatest degree of suppressed burning was exhibited by this propellant. Thus the additive operates to increase the melt viscosity, impeding the interference of the fine AP burning and restoring normal burning where it would otherwise be suppressed. At higher pressures normal burning was restored and the fine AP burnt ahead of the coarse AP, leaving protruding coarse AP particles surrounded by binder.

## 2.4 Dimeryl Diisocyanate for production of Bi-Plateau Propellant

Investigations of melt flows[7, 14, 24] formed the basis for determination of the effect of binder-curative system on plateau burning in composite propellants. Using hot stage microscopy an HTPB-DDI binder system was observed to melt at 260°C to a low viscosity fluid, well below the vaporisation temperature (approximately 500°C). Other curatives used with HTPB, ranked in order of melt state temperature, were toluene diisocyanate (TDI, 300°C), IPDI (330-370°C) and methyl diphenyl diisocyanate (MDI, 400°C). The large difference in melt and vaporisation temperatures were assumed to indicate relative thickness of the binder melt layer in composite propellants.

Tests for pressure deflagration limits showed that no-burn domains existed below a low pressure limit, as expected, and additionally in a mid-pressure range. The mid-pressure no-burn state was a condition related to fine AP particle size, low oxidiser/fuel ratio and a high degree of binder melt. The effect of binder curative was significant. The HTPB-DDI binder system exhibited the most melt flow. The AP/HTPB-DDI propellant which managed to sustain burning did so in a narrow pressure range (1.5 – 3 MPa) only. The AP/HTPB-IPDI system featured a well defined non-burning domain in the mid-pressure range. It was concluded that the ability of the HTPB binder to melt could be tailored by choice and combination of curing agent(s).

More severe anomalous combustion and lower burn rates were observed in wide oxidiser particle size distribution propellants containing DDI than those containing IPDI[24]. Boxx et al.[25] found IPDI was more effective in restoring normal burning than DDI and assumed that this indicated an ability of the matrix to burn ahead of the coarse AP. Formulations containing DDI exhibit bi-plateau burning, an additional plateau forming at low pressure (1.4 – 4.8 MPa)[6, 3, 4]. Liquefaction of the DDI cured binder has a lower activation energy[43] and takes place more rapidly than binder cured with IPDI.

The essential difference in HTPB cured with DDI compared to that cured with IPDI is related to the cleavage of the urethane linkage [22, 24, 32, 43, 44, 45]. The decomposition of IPDI cured binder involves gradual cleavage and liquefaction and is coupled with diisocyanate volatilisation. The urethane cleavage in DDI cured binder is not associated with significant volatilisation until higher temperatures, such as those associated with IPDI, are reached [3, 43]. Chakravarthy et al. [24] and Chakravarthy [45] assumed that the different positions of the -NCO groups in the cured binder was significant. They state that for DDI, the groups are symmetrically positioned whilst with IPDI, they are unsymmetrical, hence a wider temperature range for cleavage of the urethane linkages with IPDI and a less decisive "melt" process.

Frederick et al. [46] studied the difference in melt layers produced in binders made using DDI and IPDI curatives. They examined the surfaces of rapidly extinguished propellants with wide AP particle size distribution and HTPB binders using SEM. The result of a DDI cured binder was a 20-30 percent reduction in propellant burn rate over the pressure range 1.7 – 13.8 MPa. A model was proposed whereby the propellant burning rate depends on the fraction of oxidiser particles covered with molten binder, especially the fine particles.



The greater the fraction covered, the further the burn rate is reduced, even resulting in total extinguishment. The value of covered area was found to be related to the fluidity of the molten binder and roughness of the burning surface. Unfortunately the technique was not found to be adequate to quantitatively determine the extent of the influence of curatives on the melt layer; however, binder flow was confirmed as a mechanism.

### 3. Tailoring Burn Rate Plateaus

#### 3.1 Formulation Variables and Their Effect on Burn Rate Plateaus

##### 3.1.1 Level of Oxidiser

An increase in the fraction of oxidiser present in the formulation is usually accommodated by reducing the amount of binder. Although the oxidiser level can vary from 65 wt% to 90 wt%, the baseline propellant contains 86 wt% ammonium perchlorate [3, 6, 35]. Freeman et al. [26] studied a range of propellant formulation variables critical to plateau burning. For monomodal AP particle size propellants, increasing the oxidiser content (increasing O/F ratio) led to a reduction in the anomalous burning which is a factor in plateau burning behaviour in bimodal propellants.

##### 3.1.2 Oxidiser Particle Size Distribution

A wide distribution oxidiser particle size must be maintained within certain limits to provide the abnormal depressed burning [12, 14, 15]. The formulation should contain fine AP with a particle size in the range 2  $\mu\text{m}$  to 5  $\mu\text{m}$  and coarse AP with a particle size from 150  $\mu\text{m}$  to 400  $\mu\text{m}$  [3]. The use of smaller coarse sizes (<150  $\mu\text{m}$ ) and larger fine sizes (>5  $\mu\text{m}$ ) produces a propellant without sufficient AP particle size distribution.

The AP fraction will normally comprise approximately 50-60% coarse and 40-50% fine particles [3]. Boxx et al. [25] employed SEM to compare features on the surface of propellants extinguished whilst burning at pressures ranging from 5 to 29 MPa. Three of the formulations tested exhibited negative (mesa) burn pressure exponents at pressures from 10.3 to 17.2 MPa. The formulations contained ammonium perchlorate particles in varying coarse/fine ratios. Wide spacing of the fine AP in the matrix produced suppressed burning, whilst closer spacing (increased fine fraction) would eventually restore normal burning at all pressures. Extremes of coarse/fine ratio promote normal burning and the cessation of the abnormal burning exhibited by wide-distribution particle size propellants.

A typical formulation (refer to Appendix A) contains coarse and fine AP in the ratio 62:38 (86 wt% AP) having coarse particles of 400  $\mu\text{m}$  and fine of 2  $\mu\text{m}$  [35, 47]. Bi-plateau results were also achieved with a coarse particle size of 200  $\mu\text{m}$ . Increasing the coarse particle size lowers the burn rate of the propellant providing a "secondary" tailoring tool [6, 35].

### 3.1.3 Level of Plasticiser

Plasticiser is normally added from about 1 to 3% by weight [3]. The baseline bi-plateau burning propellant formulation usually includes 1 wt% of the plasticiser, dioctyladipate (DOA). Increasing the level of plasticiser reduces the viscosity of the binder melt layer, enhancing the abnormal burning behaviour [37] and producing a more defined plateau [3].

### 3.1.4 Level of Burn Rate Modifier

A wide AP particle size distribution propellant produces severely depressed abnormal burning at intermediate pressures. Although a number of refractory metal oxides may be selected as a burn rate modifier, titanium dioxide is preferred to restore normal burning whilst best allowing tailoring of the plateau or mesa [3, 6, 35].

For most applications, the preferred refractory oxide content in the propellant will be in the range 0.3% to 5% [6, 35, 47]. As the level of titanium dioxide increases so does the burn rate whilst the pressure exponent decreases. Later work refined the formulation and suggested 1.5 – 2.0% as more appropriate [3] as lower levels of titanium dioxide decrease plateau definition. The addition of 2% titanium dioxide produced a more distinct plateau [6, 35] and can be regarded as the amount added for a baseline formulation (see Table 2).

### 3.1.5 Particle Size of Burn Rate Modifier

Particles of titanium dioxide added to the propellant with a size between 0.02  $\mu\text{m}$  and 0.8  $\mu\text{m}$  will produce a plateau [6, 35]. A particle size of 0.4  $\mu\text{m}$  is considered to provide a useful plateau as a baseline. The larger particle sizes were found to perform better at low burn rate ranges and small particles at the higher burn rates [6, 35]. A blend of 0.02  $\mu\text{m}$  (0.5 wt%) and 0.4  $\mu\text{m}$  (1.5 wt%) particles produced a bi-plateau propellant with exponents approaching zero in both the low and high pressure ranges and burn rates slightly higher than the baseline propellant [6, 35] (2 wt% 0.4  $\mu\text{m}$ ). Blending of titanium dioxide particle sizes would thus enable tailoring of the burn rate levels.

### 3.1.6 Type of Curative

To produce bi-plateau burning, DDI is added as the curative; in conventional AP/HTPB propellants this leads to lower burn rates and pressure exponents [44]. Two pressure regions separated by an intermediate region with high exponent as exhibited by a formulation containing DDI are presented in Figure 4 (labelled Propellant 1). When IPDI is used as curative, a single plateau at intermediate burn rates is produced (see Propellant 2 in Figure 4).

Blending of curatives would allow tailoring of the burn rate levels. As more IPDI is substituted for DDI in the formulation the burn rate of the high pressure plateau increases, whereas the low pressure plateau is inclined to be eliminated [6, 35].

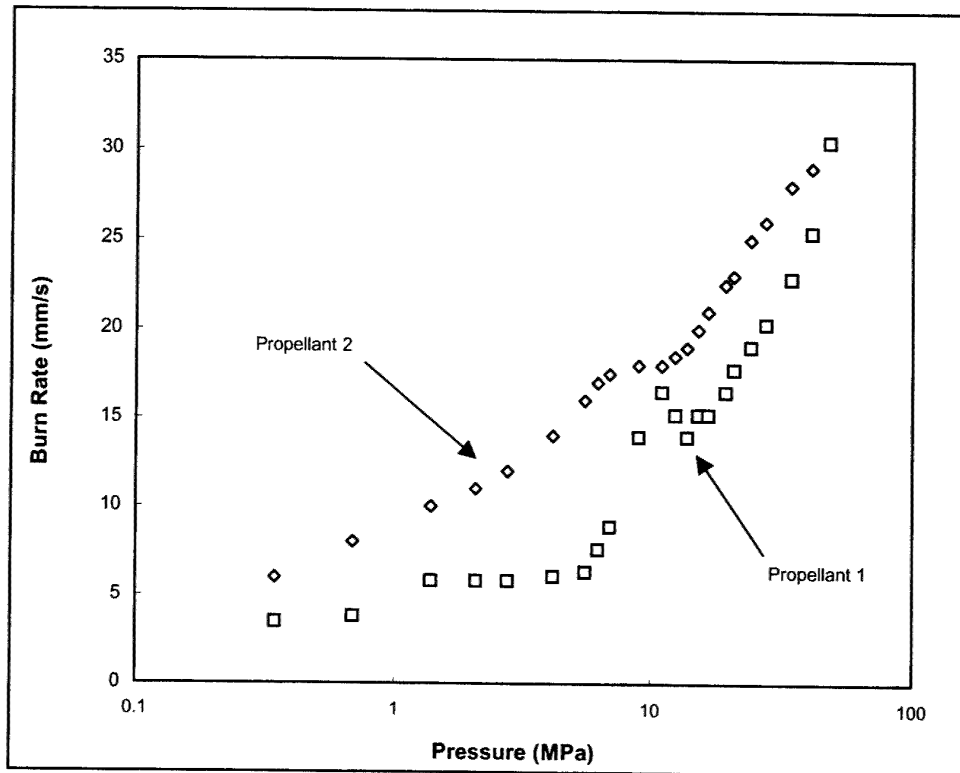


Figure 4 Chart showing effect of curative type on plateau burning

### 3.1.7 Metal Fuel

Various studies have reported the approach to production of bi-plateau burning propellants which include a metal fuel [3, 6, 35, 47]. The preferred metal is aluminium but magnesium or other suitable metals may be employed. Generally the aluminium content will vary between 5 and 25 wt% with 15 wt% being common. The particle size of the aluminium affects the plateau burning behaviour. A large particle size from 80-120  $\mu\text{m}$  is preferred.

### 3.1.8 Other Additives

A number of other ingredients may also be included in the formulation in trace amounts as processing aids and to confer more desirable characteristics on the cured propellant [3, 6, 35]. Due to the high solids loadings in bi-plateau propellant formulations, aids to attaining complete mixing are important. Tepanol (HX-878), a 3M product, is a bonding agent and processing aid. It reacts with the surface of the AP, reducing the end of mix viscosity by the increased wetting of solid ingredients. Consequently, the cured propellant has improved mechanical properties due to better bonding between binder and oxidiser.

The reaction of Tepanol with AP liberates ammonia which must be removed by vacuum prior to addition of the curative. Octadecylisocyanate (ODI) is added to scavenge ammonia liberated by the Tepanol reaction and not removed by vacuum as a processing aid. Triphenyl Bismuth (TPB) is a cure catalyst added to ensure complete cure in a timely manner.

### 3.1.9 Energetic Binders

The basic formulation for a bi-plateau propellant includes a non-energetic binder. Hydroxy-terminated polybutadiene is used in the baseline formulation, however, other binders such as polyether, polyester and PBAN are also suitable. One report suggested that energetic binders such as polyglyn (PGN) or energetic oxetane binders may be acceptable in certain situation(s) but that the effective plateau could be reduced[3]. The use of energetic binders allows reduced solids loadings, meaning an improvement in the insensitiveness of the propellant. Campbell[4] reported the bi-plateau burning of a composite propellant containing poly(nitratomethylmethyloxetane) (polyNMMO) in the baseline formulation. Other suitable binders were poly(azidomethylmethyloxetane) (AMMO) and poly(bisazidomethylmethyloxetane)-copoly(azidomethylmethyloxetane) (BAMO-AMMO). The inclusion of an energetic plasticiser, such as triethylethylene glycol dinitrate (TEGDN) or butyl (nitratomethyl) nitramine (BuNENA) in preference to a non-energetic one, compliments the energy separation between binder and solids.

As described above, in an AP/HTPB propellant, it was necessary to use DDI to produce bi-plateau behaviour [4]. The use of DDI with polyNMMO leads to elimination of the low pressure plateau and significant reduction of the high pressure plateau. The use of Desmodur N-100 yields bi-plateau burning behaviour, while IPDI increases the burn rate at low pressure and improves the plateau at high pressure. Triphenyltin chloride (TPTC) may be included as a cure catalyst and methyl-p-nitroaniline (MNA) as a stabiliser. Titanium dioxide of the same particle size and quantity as used in AP/HTPB propellant must be added as the burn rate modifier. A bimodal blend of AP with similar wide-distribution particle size must also be maintained. The factors affecting the plateau locations are the same as those in AP/HTPB formulations, refractory oxide and AP particle size, distribution and content, plasticiser content and cure agent selected.

## 4. Conclusion

This paper reviews the literature available to the author up to August 2001 on the mechanism of combustion in bi-plateau burning composite propellant. Some information was unavailable because of classification or export controls.

The essential factors for producing bi-plateau burning behaviour in composite AP/HTPB propellants are:

- 1) A wide distribution of AP particle size is foremost. Coarse particles above 150  $\mu\text{m}$  and fine below 5  $\mu\text{m}$  are required. These sizes must exist in a definite ratio to ensure adequate interstitial spacing. This combination promotes abnormally depressed burning due to interference of the fine AP fraction combustion by the binder melt layer [12, 20, 14, 15].
- 2) Inclusion of a burn rate modifier, preferably titanium dioxide, in the formulation, which acts to increase the binder melt layer viscosity restoring normal burning (by preventing binder melt interference) but only at low and high pressures, leaving abnormal burning and a plateau or mesa effect at intermediate pressures [6, 25, 26, 35, 47].
- 3) Use of DDI curative. DDI produces a plateau at lower pressures due to increased binder liquefaction [7, 14, 24, 37, 46].

The existence of melt layer interference was established as the mechanism leading to plateau burning. Most workers presented arguments for titanium dioxide restoring normal burning at low and high pressures by acting to increase the viscosity of the binder melt [25, 26, 37]. It is not clear whether titanium dioxide also catalyses the decomposition of ammonium perchlorate. Whilst several studies found no catalytic behaviour, one showed evidence of an increased rate of mass loss and concluded that an adequate model for the mechanism of plateau burning in propellants containing titanium dioxide had not yet been developed [26].

Formulation variables can be adjusted to tailor the pressure levels and burn rates of the plateaus formed [6, 35, 47]. Blending of titanium dioxide particle sizes produced propellant with slightly higher burn rates (compared to a baseline formulation, see Appendix A) and exponents in the plateau regions approaching zero. Coarse particles aided formation of plateaus at low pressure and fine particles at high pressure.

The oxidiser level and particle size distribution will affect the propellant burn rate[3]. High levels of ammonium perchlorate restore normal burning, low levels reduce the size of the plateau. The particle size of the coarse fraction was described as a secondary tool for tailoring the burn rate. Increasing the coarse AP particle size decreases the burn rates. Extremes of coarse to fine ratio promote normal burning.

The level of plasticiser and type of curative used were found to affect the extent of the binder melt layer. Increasing the plasticiser produced a more defined plateau [3, 37]. When DDI was substituted for other curatives a low pressure plateau formed [3]. Blending of curatives was recommended to produce higher burn rates.

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## Appendix A: Formulations for Bi-Plateau Burning

*Table 2. Baseline Formulation for Bi-Plateau Burning AP/HTPB Propellant*

Component	Ingredient	Content (wt%)
Binder	HTPB	~8.80
Oxidiser	AP fine	32.68
	AP coarse	53.32
Curative	DDI	~2.11
Burn Rate Modifier	TiO <sub>2</sub>	2
Processing Aid	ODI	0.02
Bonding Agent	Tepanol	0.05
Plasticiser	DOA	1
Cure Catalyst	TPB	0.02

*Table 3. Baseline Formulation for Bi-Plateau Burning Energetic Oxetane Propellant*

Component	Ingredient	Content (wt%)
Binder	PolyNMMO	~15.02
Oxidiser	AP fine	32.90
	AP coarse	37.10
Curative	N-100	~0.72
Burn Rate Modifier	TiO <sub>2</sub>	2
Stabiliser	MNA	0.25
Cure Catalyst	TPTC	0.01
Plasticiser	BuNENA	11.0
Metal Fuel	Al	1

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## 19. ABSTRACT

The literature describing the mechanism of combustion in composite rocket propellants exhibiting bi-plateau burning has been reviewed. A wide distribution of AP particle size in a definite coarse/fine ratio is required to ensure adequate interstitial spacing resulting in abnormally depressed burning due to interference of the fine AP fraction combustion by the binder melt layer. Titanium dioxide added to the formulation acts to increase the binder melt layer viscosity restoring normal burning at low and high pressures, leaving abnormal burning and a plateau or mesa effect at intermediate pressures. The use of dimeryl-diisocyanate curative produces a unique plateau at lower pressures due to increased binder liquefaction. Formulation variables can be adjusted to tailor the pressure levels and burn rates of the plateaus formed.

